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BY THE POLAROGRAPHIC METHOD

by  
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# DETERMINATION OF SMALL AMOUNTS OF URANIUM BY THE POLAROGRAPHIC METHOD

By Scott B. Kilner

There are two analytical problems in the purification of the uranium. The first is the determination of small amounts of uranium in the ferric hydroxide precipitate, and the other is the examination of the solution remaining after the precipitation of the ammonium uranate for any unprecipitated uranium. In both of these cases any uranium will be present in very small amounts, and will have a large excess of other ions.

A method has been published<sup>1,2</sup> for determining uranium in the ferric hydroxide precipitate polarographically, which is claimed to be sensitive to 0.1% uranium in ferric hydroxide. It has been found possible to determine approximately 50  $\mu\text{m}/\text{cc}$  of uranium, and by using enough ferric hydroxide to give a final solution nearly saturated with ferrous chloride, it should be possible to detect as little as one part in ten thousand of uranium in ferric hydroxide and estimate it to within approximately 20%. The presence of 10  $\mu\text{m}/\text{cc}$  of uranium can be noticed if the run is carefully made, which means that one thousandth of a per cent of uranium can be detected in the ferric hydroxide precipitate.

The method is reasonably rapid. A complete determination starting with the hydroxide can be completed in approximately one hour, of which most of the time is spent in waiting for the oxygen to be flushed out of the sample solution.

The problem of determining how much, if any, uranium remains after the ammonia precipitation is much the same as the preceding.

## PROCEDURE

a) Ferric hydroxide is dissolved in 0.1N HCl which is 2N in hydroxylamine hydrochloride to give a solution of approximately 80 g/100 cc of ferric hydroxide. After boiling for 2 to 3 minutes to reduce the ferric iron (which interferes) to ferrous, continue as in c.

b) The solution from the ammonia precipitation is evaporated down until salts start to come out when cold, then reduced to about half this volume, and diluted with an approximately equal volume of 4N hydroxylamine hydrochloride which is 0.2N in HCl. Boil for 2 to 3 minutes to reduce the iron, and proceed as in c.

c) The solution prepared as in a or b is placed hot into the electrolysis vessel, and washed with oxygen-free nitrogen or with hydrogen for approximately one half of an hour to remove air. It is very important to remove every trace of oxygen, as one of the oxygen waves comes out at approximately the same potential as uranium, and might give spurious indications of the presence of uranium.

A polarogram is made from the sample in the usual manner, a known increment of uranyl solution added, which will about double the uranium concentrations, and a second curve made.

This method of increments is necessary since each sample is different, and both the diffusion current and the half-wave potential of uranyl ion depend on the pH and salt concentration, making it difficult to standardize the instrument.

The method as it stands is a very sensitive method for the rough estimation of uranium.

1. Strubl, R., *Collection Czechoslovak Chem. Commun.* 10:466 (1938).

2. Kolthoff, I. M. and J. J. Lingane, *Polarography*, p293, Interscience Publishers, Inc., New York, N. Y., 1941.